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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant(s):	Clark, Steven A. Pillai, Balathandan S.
Title:	METAL ALLOY PRODUCT AND METHOD FOR PRODUCING SAME
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Examiner:	Combs-Morillo, Janell A.
Art Unit:	1742
Attorney Docket No.:	043420-0118

#13

Commissioner for Patents
Washington, D.C. 20231

DECLARATION

I, Arvin Montes, Ph.D., declare and say as follows:

1. I submit this declaration in support of the above-identified application for a U.S. patent and in support of the Applicants' response to the Final Office Action issued on February 7, 2003 (the "Final Office Action").

2. I received a B.Sc. in Mechanical Engineering from Marquette University in 1994 and a Ph.D. in Materials Science Engineering from Marquette University in 2001. For the past two and one-half years, I have been employed as a Senior R&D Materials Science Engineer at Johnson Brass & Machine Foundry, Inc. My professional experience has involved work related to the development, manufacturing and/or testing of materials made from a variety of cast and wrought alloy chemistries, particularly centrifugally cast aluminum alloy materials.

3. In preparing this declaration, I have reviewed the above-identified patent application, the claims currently pending in the application, the Final Office Action issued on February 7, 2003, and the U.S. patents and other references cited in the Final Office Action.

4. In my opinion as one skilled in the metallurgical art, the elongated grain structure observed in wrought alloys produced by mechanical working techniques is distinctly different from the "generally round grain structure" (i.e., equiaxed grain structure) that is observed in alloys produced by the method described in the present application. I interpret the phrase "generally round grain structure" as used in the present application to refer to the three-dimensional shape of the grain structure of an alloy material, i.e., to an alloy material which has a substantially equiaxed microstructure. Such a material would be expected to exhibit the equiaxed grain structure when its microstructure is viewed in cross section along all of the major axes of the structure (longitudinal, long transverse or short transverse). The use of the term "generally round" to characterize the general three-dimensional shape of the grain structure is similar to the characterization of grain structure as "spherical" used in one of the references cited in the Final Office Action (see U.S. 6,120,625 ("Zhou")).

5. Centrifugally cast aluminum alloys differ from that of the cast ingot microstructure in that the microstructure consists of uniform equiaxed grains (see Figure 1). The result of the uniform equiaxed grain structure formed by centrifugal castings has been summarized by Dobson: "Centrifugal castings are best described as isotropic, that is, having equal properties in all directions. This is not true of a forging." (ASM Handbook, Volume 15, Casting, 1988, pg. 300).

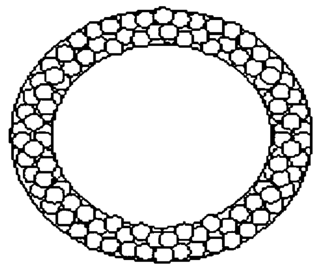


Figure 1. Schematic of the equiaxed grain structure of an aluminum centrifugal casting.

6. In the present application, the phrase "generally rounded" is used to describe the grain structure of a centrifugally cast aluminum product. A typical aluminum centrifugal casting consists of a fine equiaxed grain structure, *i.e.*, grains having substantially equal length in all directions. In the present application, I would interpret the phrase "generally rounded" to mean that the grain structure is equiaxed. In addition, the materials produced by the present method exhibit isotropic mechanical properties, *i.e.*, equal properties in all directions/orientations (see data and discussion below). Materials that exhibit isotropic properties typically have an equiaxed grain structure.

7. Exhibits A-D attached hereto show the microstructure of four different aluminum alloys having wrought chemistries. The alloys were produced by a process which includes centrifugal casting and subsequent hipping of the alloy material. The microstructures shown demonstrate the equiaxed grain structure of these materials. All four aluminum alloy materials shown in Exhibits A-D were produced by the present method and have a substantially uniform and generally round grain structure, i.e., a substantially uniform and equiaxed grain structure.

8. In contrast to the aluminum products produced by the present method, wrought worked aluminum products typically exhibit anisotropic mechanical properties, *i.e.*, the dependence of mechanical properties on orientation. Anisotropic mechanical properties can also be described as "directional properties" (Hatch, J.E., Aluminum:Properties and Physical Metallurgy, ASM International, 1984, p. 375-377; "Hatch") or "preferred orientation" (Dieter, G.E., Mechanical Metallurgy, 3rd Ed.. McGraw–Hill Publishing Company, New York, 1986, p. 322-324; "Dieter"). The variation in mechanical properties of wrought materials and their dependence on orientation can be summed up by Dieter's description of *preferred orientation*:

> "It is frequently found that the tensile properties of wrought-metal products are not the same in all directions. The dependence of properties on orientation is called *anisotropy*. Two general types of anisotropy are found in metals. *Crystallographic anisotropy* results from the preferred orientation of the grains which is produced by severe deformation. Since the strength of a single crystal is highly anisotropic, a severe plastic deformation which produces a strong preferred orientation will cause a polycrystalline specimen to approach the anisotropy of a single crystal. The yield strength, and to a lesser extent the tensile strength, are the properties most affected."

9. As described by Dieter, anisotropic mechanical properties are related to the grain structure of the alloy. Hatch further states that mechanical properties "are governed by the section thickness or the cross-sectional area of the wrought product of the alloy selected and, of more importance, by the direction in which the test was made." During the wrought process, grains become elongated in the direction of working. The metallurgical art provides many examples that illustrate the production of an elongated grain structure in metal alloys produced by wrought working techniques. For example, Polmear, I J. "Light Alloys: Metallurgy of the Light Metals" (Edward Arnold (Publishers) Ltd.; "Polmear") contains a discussion of the effect of mechanical working on wrought aluminum alloys. At page 32, Polmear states that "[m]ost wrought products do not undergo bulk recrystalization during subsequent heat treatment so that the <u>elongated grain structure</u> resulting from mechanical working is retained." (emphasis added) A copy of this excerpt from Polymear has previously been submitted to the file for this application.

10. Typically, the nomenclature of the axes is related to the direction of working, as shown in Figure 2 (taken from Chu, H.P. and Wacker, G.A., "Fracture Toughness and Stress Corrosion Properties of Aluminum Hand Forgings," 1972, *Journal of Materials*, JMLSA, Vol. 7, No. 1, March, pp. 95-99; "Chu"). Figure 2 depicts a composite picture of a 6061-T652 hand forging consisting of microstructures taken in three different orientations. The directional effects are generally measured in planes or directions, as described by Hatch:

> *Longitudinal:* Parallel to major dimension or directions of working of section.
> *Long Transverse:* 90° to direction of working and parallel to width of section.
> *Short Transverse:* 90° to direction of working and parallel to thickness or minimum dimension of section.
> *Transverse:* 90° to direction of working in product having axial symmetry.

The following nomenclature is used for Chu's 6061-T652 hand forging: longitudinal (R), long transverse (W), and short transverse (T). For wrought aluminum sheet, mechanical properties are reported in the following directions: longitudinal (L), long transverse (T), and short transverse (S). For wrought aluminum extrusions, mechanical properties for longitudinal and transverse directions are reported.

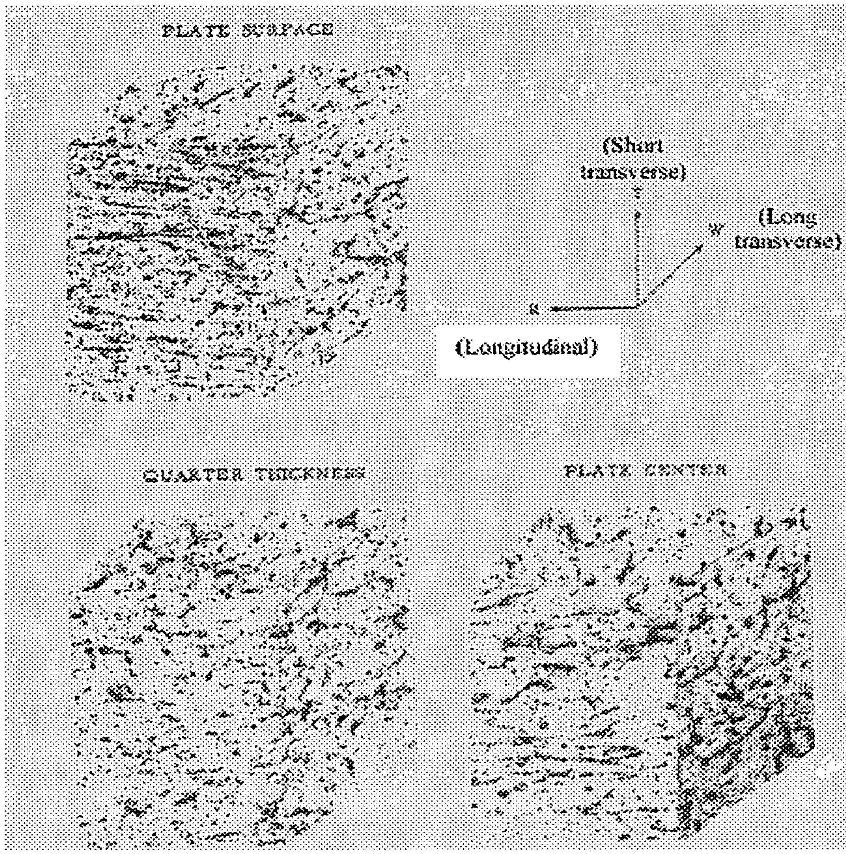


Figure 2. Composite photomicrographs illustrating structural variations from surface to center of 9-in.-thick 6061-T652 hand forging.

11. The material shown in Figure 2 could easily be mistaken as having an equiaxed grain structure, *i.e.*, equal dimensions in all directions, if the microstructure were viewed exclusively in the RW plane. Observation of the microstructures in both the TR and TW planes, however, clearly shows the material to have an elongated grain structure. In order to determine if the grain structure of a material is equiaxed, microstructures of the material must be viewed in more than one orientation or direction. ASM Handbook, Volume 9: Metallography and Microstructures, Pg. 357, states the following regarding the determination of grain size:

> "Because grains are seldom completely equiaxed in most wrought alloys, they must be measured in three dimensions using

> standardized section planes, and require some auxiliary expression of grain shape."

The grain structure of the material shown in Figure 2, and in the case of other wrought materials, has a profound effect on the mechanical properties of the material. Chu notes that significant anisotropy is present in the hand forgings studied. In terms of mechanical properties, Chu states the following:

> "The mechanical properties depended significantly on the directionality of the forgings (Table 2). Both strength and ductility were lower in the short transverse direction than in the longitudinal or long transverse direction. There were also some variations in strength and ductility from the surface to the center."

Chu's observations are supported by the following statement made by Hatch, "typically, tensile strengths are higher in the longitudinal direction than in the long transverse direction."

12. Claims 33-39, 46-52, and 54-58 were rejected in the Final Office Action based on Shaffer *et al.* (U.S. 6,248,189 :"Shaffer") and ASM Handbook Vol. 9 Metallography and Microstructures" pp. 357-388 (hereinafter "ASM Handbook Vol. 9). The grain structure of the centrifugally cast aluminum products produced by the present method differ from wrought products in that the grains are equiaxed. The present centrifugally cast products are able to obtain the equiaxed grain structure by omitting the working processes involved in the wrought process. Using the centrifugal process, however, allows the production of a material having a uniform, fine, equiaxed grain size, which ultimately results in mechanical properties that meet those of its wrought counterparts. In addition, mechanical properties are isotropic, *i.e.*, independent of orientation or direction.

13. In Shaffer, "the invention provides an aluminum alloy having a uniform fine grain structure which allow such extruded tube to be drawn without the need for an expensive separate furnace heat treatment" (Column 3, Lines 30-34). Shaffer's use of the phrase "uniform fine grain structure" does not imply an equiaxed grain structure, but simply states that the grains are approximately the same size and shape. Shaffer reports that samples used to determine grain size "were evaluated along the length" (Column 5, Line 67). According to ASTM E112-88, the

average grain diameter of a deformed structure should be calculated using the following equation:

Average grain diameter = $3(dL^{-1} + dS^{-1} + dT^{-1})^{-1}$, where
L = logitudinal,
S = short transverse, and
T = long transverse.
If dT = dL, as in the case with most extrusions, then
Average grain diameter = $3(2dL^{-1} + dS^{-1})^{-1}$.

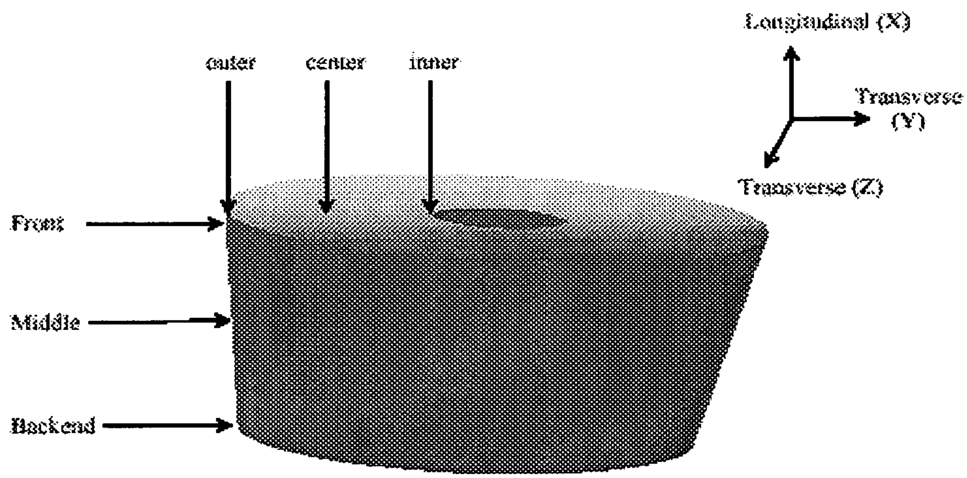


Figure 3. Schematic of Shaffer's grain size analysis of extruded 6XXX aluminum.

14. Since the results reported in Shaffer are based on just one direction, "along the length," uncertainty exists as to whether the grain structure is equiaxed. As stated in ASM Handbook, Volume 9: Metallography and Microstructures, Pg. 357, "some auxiliary expression of grain shape" is necessary for wrought products. Figure 3 represents a schematic of an extruded part and the locations analyzed by Shaffer. Shaffer does not, however, state the orientation of the sample analyzed at each location. In order to determine if the grain structure is equiaxed, microstructures must be prepared in the YZ and the XY (or XZ) planes for each location.

15. Figure 4 is a common grain structure found in extruded aluminum (see also Exhibit E). The X-axis denotes the longitudinal direction or the direction of extrusion. Using micrographs from the YZ plane alone, Figure 2 of Exhibit E, the material appears to possess an equiaxed grain structure. As Figure 1 of Exhibit E clearly shows, however, the material possesses an elongated grain structure. As a comparison to the work performed by Shaffer, the phrase "along the length" does not clearly describe the orientation used for the grain size analysis. Shaffer does not report the grain structure to be equiaxed, nor does he provide an "auxiliary expression of grain shape." Section 11.6.2 of ASTM E112 summarizes the notation, in the form of subscripts, used for non-equiaxed grain structures of wrought products. One skilled in the art of metallurgy would expect a wrought product to have an elongated structure in the direction of working. In addition, claims of an equiaxed grain structure as a result of recrystallization must be supported by microstructures and/or grain size measurements in two different orientations. Since Shaffer fails to provide any evidence of an equiaxed grain structure, the wrought extrusion would be assumed to contain an elongated grain structure.

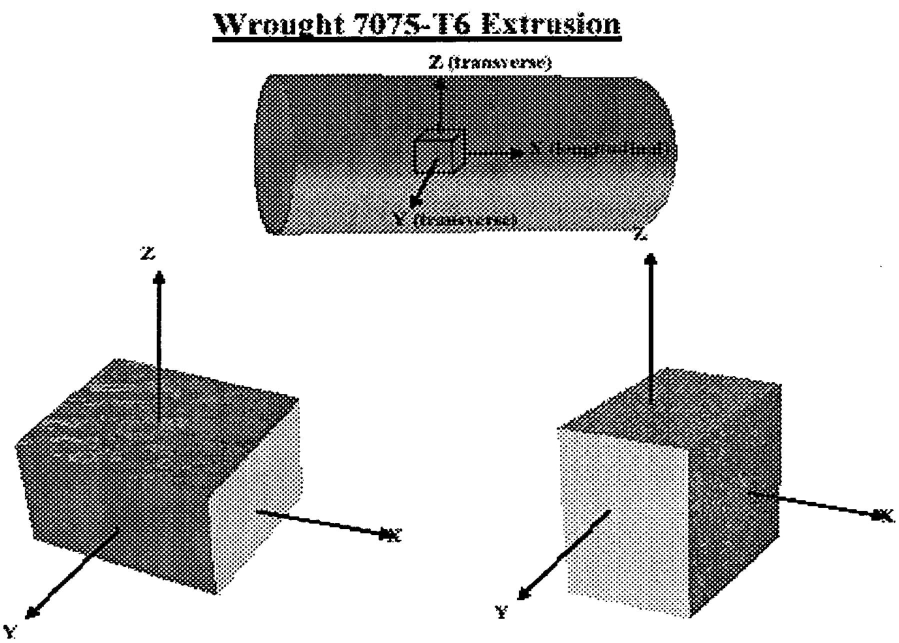


Figure 4(a). Microstructure of 7075-T6 extrusion in the XY plane.

Figure 4(b). Microstructure of 7075-T6 extrusion in the YZ plane (direction of extrusion).

16. An alternative method, although less effective, of determining if the material possessed an equiaxed grain structure, would be to compare mechanical property results in two different orientations or directions. If the material were to contain an equiaxed grain structure, its mechanical properties would be expected to be identical in all orientations or directions. For wrought extrusions, mechanical properties are typically reported in the longitudinal direction only, due to size limitations. As in the case with Shaffer's extruded material, mechanical property results are limited to the longitudinal direction due to the size of the extrusion (4.500" O.D. by 0.087" wall thickness). An example of the variation in grain size and shape with orientation or direction can be found in "ASM Handbook Vol. 9 Metallography and Microstructures," pp. 357- 388 (hereinafter "ASM Handbook Vol. 9). Figures 192 and 193 of this reference (shown in Figure 5 of this document) show an example of a 6061-T6 extrusion. In Figure 192, the extruded tube appears to have an equiaxed grain structure. The term "extrusion direction vertical" states that the extrusion direction is perpendicular to the micrograph shown. The micrograph shown in Figure 192 is analogous to the micrograph shown in Figure 4(b), *i.e.*, the YZ plane. Figure 193 is a micrograph of an assembly that includes the extruded material shown in Figure 192. The microstructure of the extruded 6061-T6 tube shows an elongated grain structure and is clearly different from that shown in Figure 192. The orientation of the extruded 6061-T6 tube is analogous to the micrograph shown in Figure 3(a), *i.e.*, the XY plane.

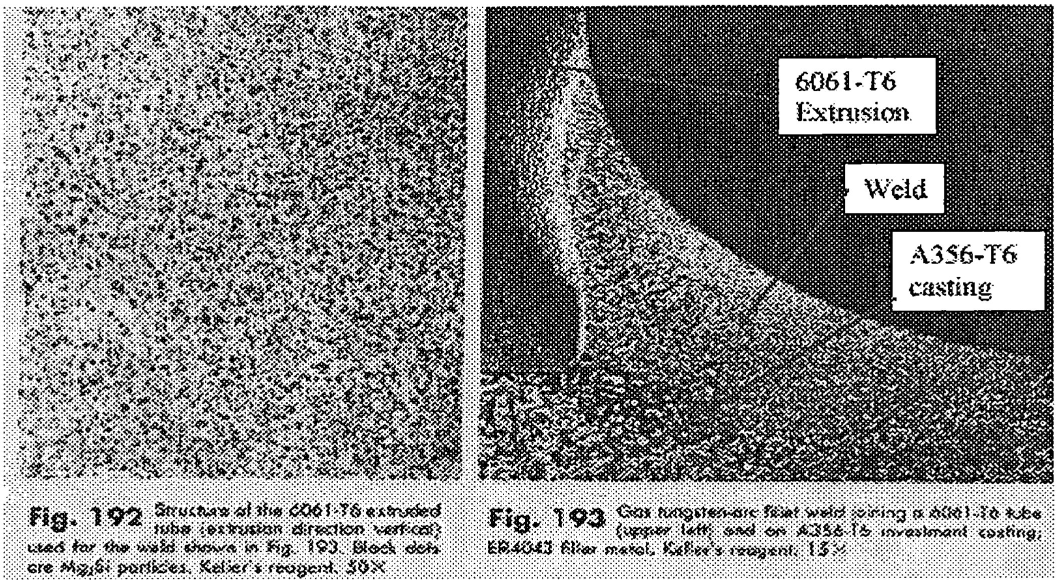


Figure 5. Microstructures from ASM Handbook, Volume 9, illustrating the need to view grain size in more than one orientation to determine grain shape.

17. The centrifugally cast aluminum materials produced by the present method exhibit a uniform equiaxed grain structure as shown in Exhibits A-D. The result of an equiaxed grain is isotropic mechanical properties of the material. The Figures in Exhibit D (JC7075-T6) also contain mechanical property data for both the X and Z directions. In the X-direction, mechanical properties are as follows: 76 ksi (tensile), 67 ksi (yield), 6.6% (elongation). In the Z-direction, mechanical properties are as follows: 77 ksi (tensile), 67 ksi (yield), 7% (elongation). Based on the microstructures and the isotropic mechanical properties, one can come to the conclusion that the centrifugally cast aluminum products made by the present method have an equiaxed grain structure, which is not typically found in wrought aluminum.

18. In summary, the claimed centrifugally cast aluminum differs from Shaffer's 6XXX extrusion based on the following observations:

1. Shaffer does not state that his material contains an equiaxed grain structure. Insufficient evidence is provided to assume that the material contains an equiaxed grain structure, *i.e.*, microstructures in two different orientations and/or mechanical property results in two different orientations.

2. ASM Handbook Vol. 9 Metallography and Microstructures clearly illustrates that an equiaxed grain structure must be determined by observing a minimum of two different orientations of the material. The 6061-T6 extruded tube shown in Figure 192 appears to have an equiaxed grain structure, but in conjunction with Figure 193, the material clearly has an elongated grain structure.

19. Claims 17, 18, 20, 21, 25, 30, 32, and 40-45 are rejected in the Final Office Action as being obvious over Kroger (U.S, 3,791,876). Kroger describes his invention as being an alloy undergoing the following steps: casting, homogenization, and fabrication into wrought forging stock, "preferably by extrusion, under specially prescribed conditions." Mechanical properties, as well as corrosion properties, of an aluminum alloy are known to be dependent on both the chemical composition and the grain structure of the alloy. The alloy material described by Kroger is significantly different than that produced by the present method. Recrystallization does not occur in centrifugally cast products and the equiaxed grain structure is a result of the centrifugally casting process. Wrought aluminum is dependent on the working process, followed by complete or partial recrystallization of new grains, to obtain the desired mechanical and corrosion properties for a specific alloy.

20. Hot working of wrought aluminum alloys is necessary in order to achieve pore closure and the formation of the recrystallized grain structure. Figure 6 illustrates the as-cast ingot microstructure prior to any working of the material. As shown by the schematic, three distinct zones exist following casting of the ingot: chill, columnar, and equiaxed. Because the ingot microstructure is not uniform and contains microporosity, the cast ingot undergoes a working process, *i.e.*, rolling, extrusion, or forging. The purpose of the hot working step can be summarized as follows:

1. Replacement of the cast grain structure (non-uniform and relatively large) with a more uniform fine grain structure through a process called recrystallization.
2. Closure and welding of any microporosity present in the cast structure.

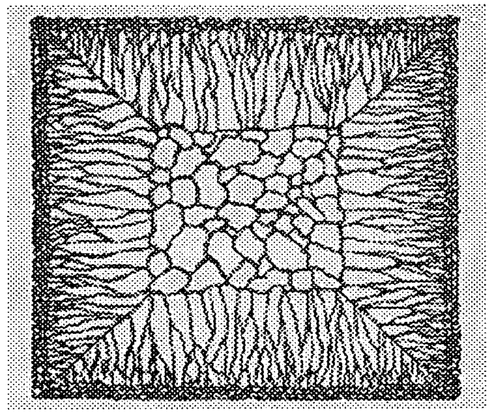


Figure 6. Schematic of cast ingot microstructure showing chill, columnar, and equiaxed zones. (Flemings, M.C., Solidification Processing, McGraw-Hill, Inc., New York, (1974).pg. 135)

21. One of the consequences of the wrought working process is anisotropic mechanical properties. As mentioned previously, mechanical properties of wrought aluminum are strongly dependent on the orientation in which the test is performed. Both rolled and forged products are tested in at least two different orientations, and typically in three orientations. Extruded products are typically tested in only one direction due to the size limitations of the product. Extruded products, however, are expected to vary in grain structure and mechanical properties in the other two directions. Variation in mechanical properties with orientation is clearly seen in ASTM, AMS, and MIL specifications that report mechanical properties in three different orientations for wrought aluminum products.

22. The claimed centrifugally cast aluminum alloys differ from that of the cast ingot microstructure, in that, the microstructure consists of uniform equiaxed grains, Figure 1 (above). The result of the uniform equiaxed grain structure can be summarized by Dobson: "Centrifugal castings are best described as isotropic, that is, having equal properties in all directions. This is not true of a forging." (ASM Handbook, Volume 15, Casting, 1988, 937 pgs.)

23. Table 1 illustrates the isotropic properties of a 7075-T6 centrifugal casting produced by the present method in comparison to the anisotropic properties of 7075-T6 wrought plate. According to the data shown in Table 1, the dependence on orientation or direction is more significant for the wrought plate. The 7075-T6 centrifugal casting does not have a strong dependence on orientation and could be considered to possess isotropic properties. The isotropic properties of the centrifugally cast 7075-T6 aluminum are directly related to the equiaxed grain structure of the material. The wrought 7075-T6 aluminum (as reported by both Hatch and Kroger) can be assumed to have a non-equiaxed grain structure due to the high degree of anisotropy in the mechanical properties.

7075-T6 Centrifugal Casting			
Test Direction	**Tensile Stress (ksi)**	**Yield Stress (ksi)**	**%Elongation (ksi)**
Longitudinal (axial)	77	67	7
Transverse (tangential)	76	67	6.6
Standard Deviation	0.71	0	0.28

7075-T6 Wrought Plate (Hatch, Pg. 377)			
Test Direction	**Tensile Stress (ksi)**	**Yield Stress (ksi)**	**%Elongation (ksi)**
Longitudinal	73.2	60.9	9
Long Transverse	71.1	58.8	4
Short Transverse	68.9	58	3
Standard Deviation	2.15	1.50	3.21
Kroger's Extruded and Forged 7075(H.S.)-T6			
Test Direction	**Tensile Stress (ksi)**	**Yield Stress (ksi)**	**%Elongation (ksi)**
Logitudinal	86	76	7
Transverse	77	66	4
Standard Deviation	6.36	7.07	2.12

Table 1. Comparison of Mechanical Properties of 7075-T6 Centrifugal Casting, 7075-T6 Wrought Plate, and Kroger's 7075(H.S.)-T6 Extrusion and Forged Material.

24. A discussion of fatigue properties has not yet been made in this Declaration, but there exists a significant difference between wrought products and centrifugally cast products. The fatigue properties of JC7075-T6 centrifugally cast aluminum produced by the present method has been shown to be equal to or superior to that of wrought plate and forgings. A hypothesis for the higher fatigue life for a given stress level is the hipping process that the centrifugally cast JC7075 aluminum underwent. During the hipping process, microporosity is removed by the diffusion of atoms to regions where microporosity exists, causing pores to shrink and eventually close. In comparison, microporosity in wrought materials is reduced in size by thermo-mechanical processing, *e.g.* hot or warm-working. Microporosity is reduced in size, but time at temperature is not long enough to promote diffusion of atoms to areas of microporosity and remove porosity completely. A more significant difference in the pore structure is the shape of the pore. The centrifugally cast JC7075 that has been HIP-ed either has no pores or a pore shape that is spherical. The pore shape for wrought 7075 products is typically similar to the grain structure, *i.e.*, elongated in nature. The pores of wrought products can act as stress risers and may serve as initiation sites for fatigue failures. Figures 7 and 8 illustrate the difference in fatigue properties between a JC7075-T6 centrifugal casting produced by the present method and 7075-T6 wrought products. For alternating stress levels lower than 30 ksi, the centrifugally cast JC7075-T6 is clearly superior to that of both the wrought forging and sheet materials. Below an alternating stress of 30 ksi, the centrifugally cast JC7075-T6 has a life 6 times longer or greater than that of wrought forgings or plate.

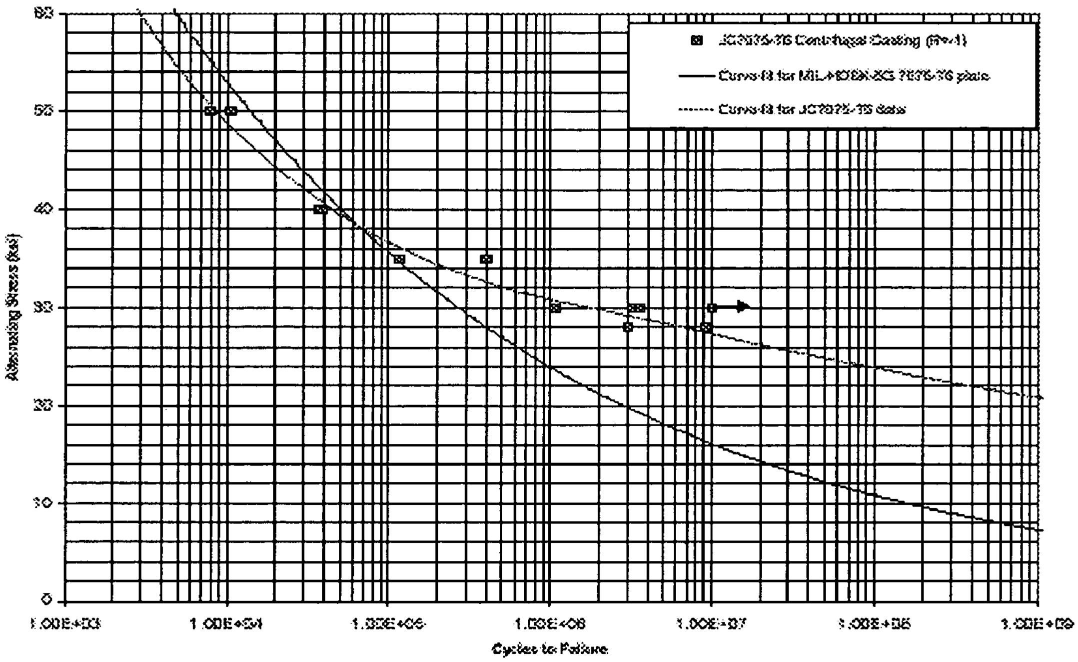


Figure 7. Comparison of Axial Fatigue Data (R=-1) for JC7075-T6 Centrifugal Casting and 7075-T6 Data from MIL-HDBK-5G (Nov. 1, 1994) (plate stock - 0.090").

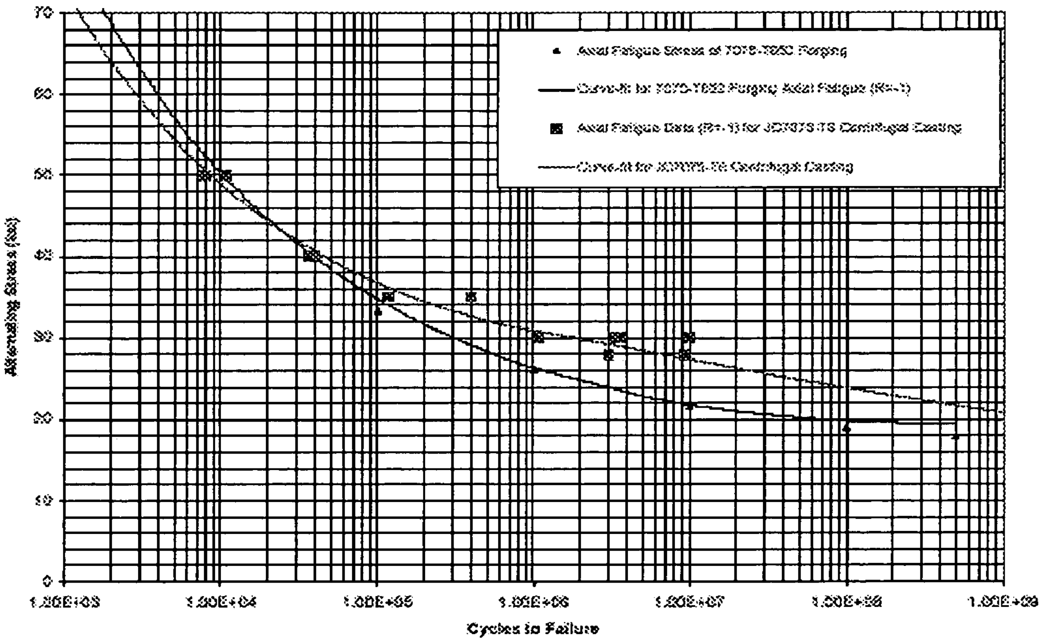


Figure 8. Comparison of Axial Fatigue Data (R=-1) for 7075-T652 Forgings and JC7075-T6 Centrifugal Casting.

25. Similar to the argument regarding the shape of the closed pore, the shape of the second phase particles ($MgZn_2$) can also play a significant role in the fatigue properties of the material. During the wrought process, second phase particles become elongated, as is the case with the grains. Due to the elongated nature of the second phase particles, these regions can act as stress risers and initiation sites for fatigue cracks. Figure 9 illustrates the elongated structure of the second phase particles in the longitudinal direction of a 7075-T6 extrusion. Figure 10 illustrates the equiaxed structure of the second phase particles of centrifugally cast JC7075-T6 material produced by the present method.

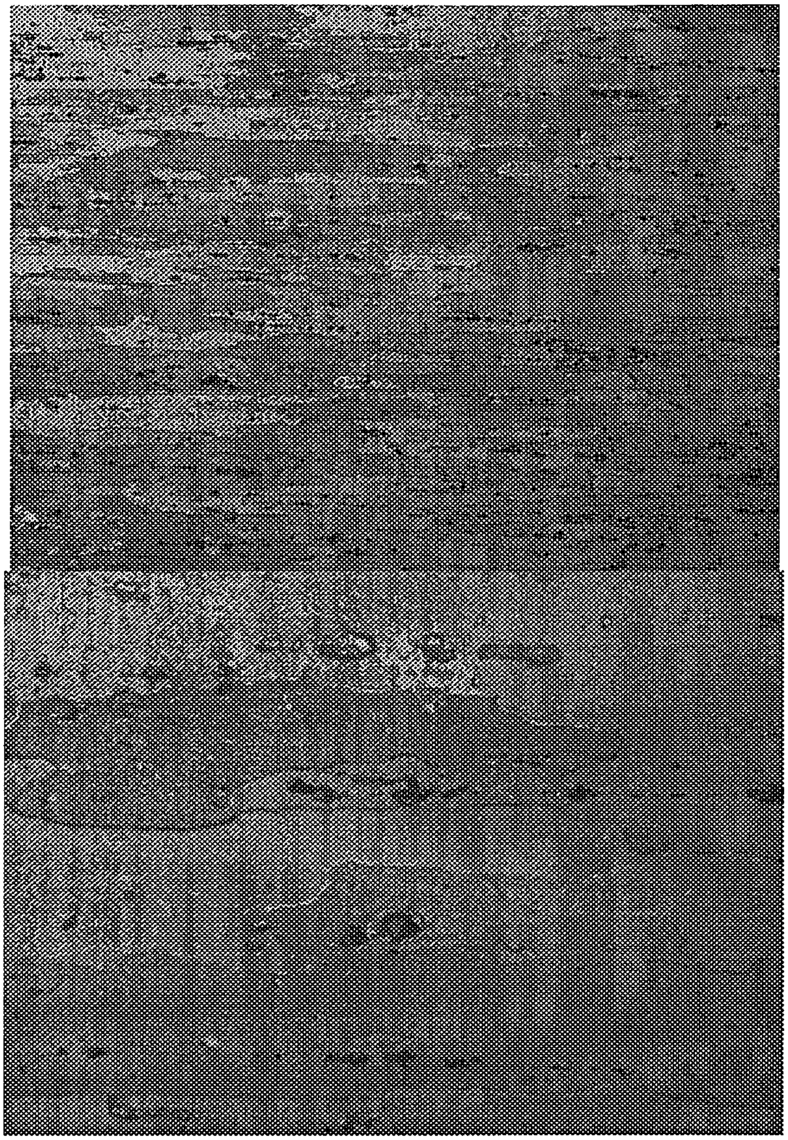


Figure 9. Microstructures of extruded 7075 aluminum at 100X (top) and 500X (bottom) illustrating the elongated structure of the second phase particles.

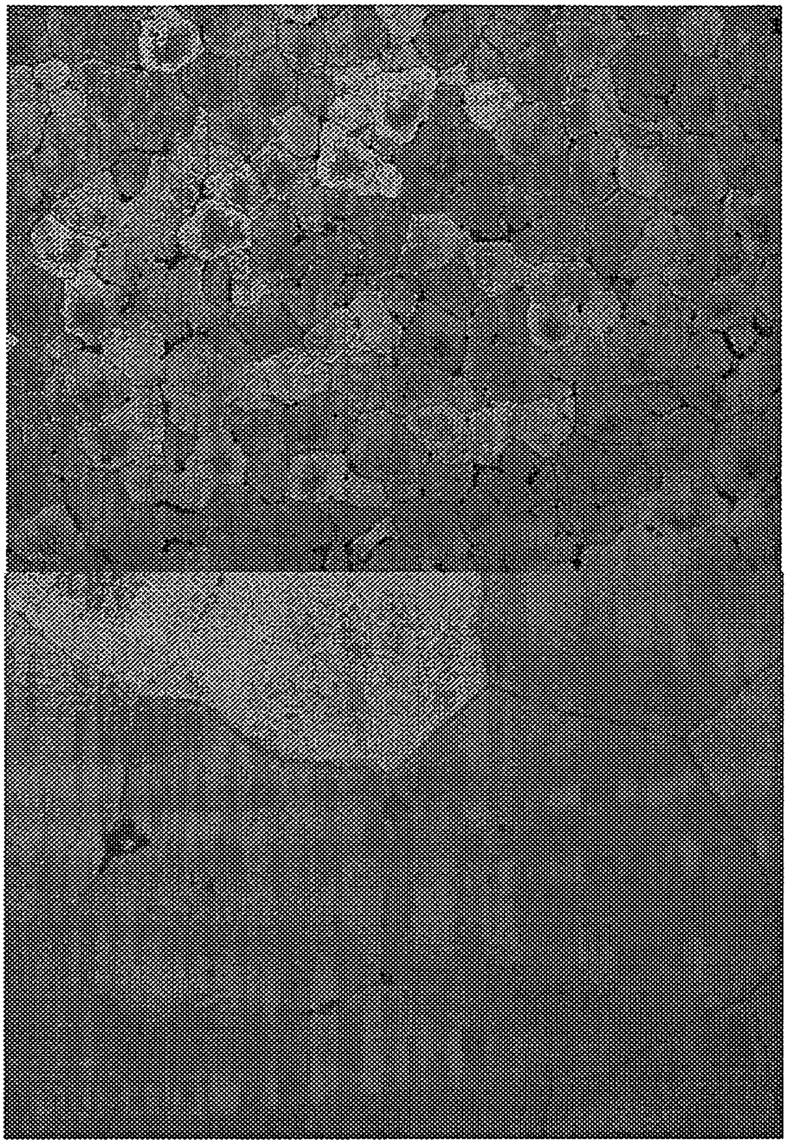


Figure 10. Microstructures of JBM's centrifugally cast JC7075-T6 at 100X (top) and 500X (bottom) illustrating the equiaxed structure of the 2 nd phase particles.

26. In summary, JBM's centrifugally cast JC7075-T6 differs from Kroger's extruded and forged 7075 based on the following:

1. The grain structure of Kroger's wrought product is not equiaxed based on the anisotropic mechanical properties shown in Table 1 (Columns 5 and 6, Lines 33-44).
2. The centrifugally cast JC7075-T6, as well as other centrifugally cast grades of aluminum, do not undergo recrystallization, but produce an equiaxed grain structure. Further evidence of an equiaxed grain structure is the isotropic properties that the materials exhibit.
3. Fatigue properties of JBM's centrifugally cast JC7075-T6 aluminum are superior to those of wrought 7075-T6 below an alternating stress of 30 ksi. The difference in fatigue properties is related to the elongated structure of closed pores and 2nd phase particles in the wrought 7075 aluminum. These elongated structures act as stress risers and initiation points for fatigue cracks.

27. Claims 17-21, 23, 24, 26, 27, 28, 32, 40-45, and 53-58 were rejected in the Final Office Action as obvious over U.S. 6,120,625 ("Zhou"). Zhou's semi-solid extrusion of AA6061 differs from the claimed centrifugally cast aluminum products based on (1) microstructure, (2) microsegregation (chemistry), and (3) mechanical properties. A comparison of microstructures between Zhou's 6061 and the centrifugally cast 6061 aluminum produced by the present method shows significant differences in the phases present and the amount of microsegregation present. In terms of mechanical properties, Zhou fails to report mechanical properties of the material. The Final Office Action contains a reference to mechanical properties associated with Figure 4(c) of Zhou. It is noted, however, that Zhou does not contain a Figure 4(c).

28. Zhou assertedly provides "a product with superior mechanical properties," but fails to produce data that supports the claim and a description of what materials are being compared. From the patent, it is unclear if he is claiming superior mechanical properties to alternative semi-solid forming techniques or to conventional wrought 6061 aluminum.

29. Zhou states that the preferred microstructure of a semi-solidly formed alloy consists of "globular or spherical grains contained in a lower melting alloy matrix" (Column 1, Lines 24-27). The claim of spherical grains is similar to that claimed in the present invention, but, the fact that the grains are suspended in a matrix of lower melting alloy makes the material significantly different. As shown in Figure 11 (Reproduction of Figure 3(a) of Zhou), the microstructure produced by Zhou consists of grains within a continuous matrix of lower melting alloy. In Figure 12 (Reproduction of Figure 3(b) of Zhou), the microstructure of Zhou's semi-solidly formed 6061 is shown, where the low melting point matrix has solidified as a dendritic structure. Both Figures 11 and 12 are significantly different from that of the JC6061-T6 aluminum produced by the present method, (see Figures in Exhibit A). The grain shapes of the two alloys are similar, however, the JC6061-T6 aluminum, as well as other grades of the centrifugally cast aluminum products produced according to this application (see additional micrographs in Exhibits B-D), does not contain a continuous network, or matrix, of lower melting point constituents. The typical, and preferred, microstructure of centrifugally cast aluminum consists of equiaxed grains and a relatively minor amount of discrete, isolated second phase particles. In other words, the alloy materials shown in Exhibits A-D have a more homogeneous grain structure and chemical composition, as compared to Zhou's material. In addition, the dendritic structure initially present in the as-cast condition, has been replaced by a more homogeneous structure following hipping and heat treatment. In contrast, Zhou's material, as shown in Figures 11 and 12, is a non-homogeneous structure containing two distinct regions.

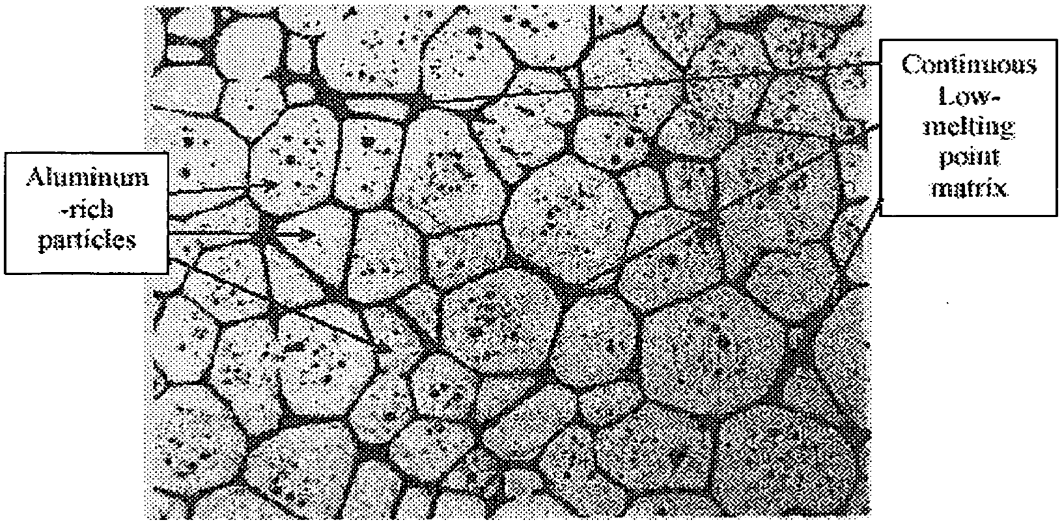


Figure 11. Zhou's Figure 3(a) showing a semi-solidly formed 6061 alloy, processed at a temperature of 620°C (1148°F). Microstructure consists of equiaxed grains (white) suspended within a matrix of low melting point alloy (black).

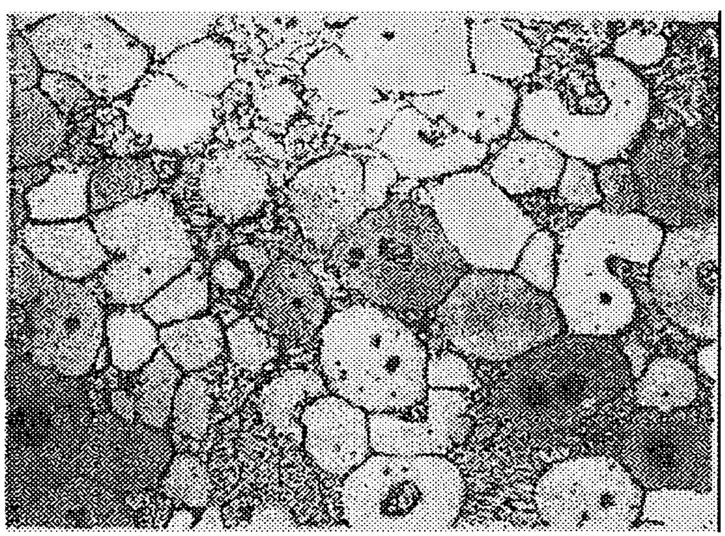


Figure 12. Zhou's Figure 3(b) showing a semi-solidly formed 6061 alloy, processed at a temperature of 630°C (1166°F). Microstructure consists of equiaxed grains and a dendritic phase that formed upon cooling from semi-solid forming temperature.

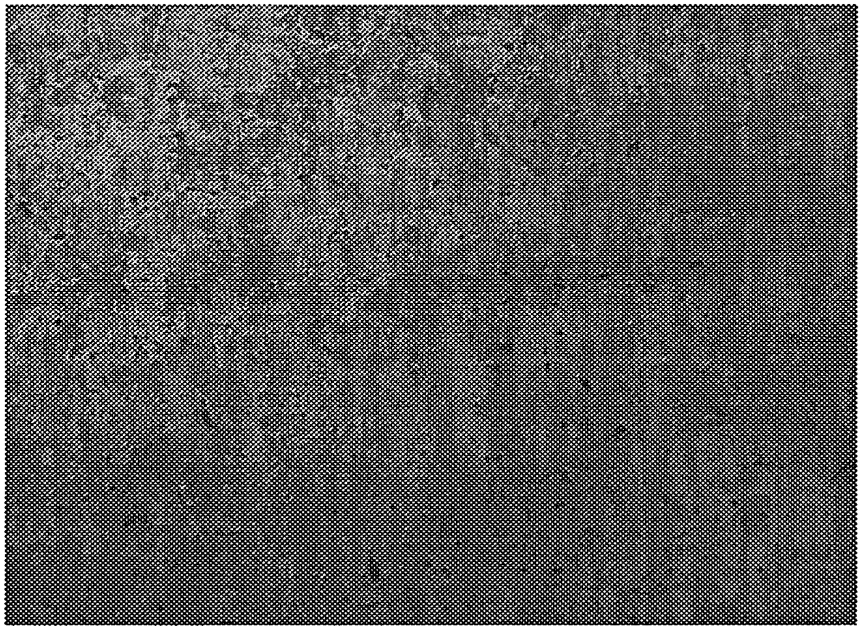


Figure 13. Microstructure of JBM's centrifugally cast JC6061-T6 aluminum alloy in the axial direction, consisting of equiaxed grains and discrete isolated 2 nd phase particles. 100X, Keller's reagent.

30. Centrifugally cast aluminum materials produced by the present method contain a more homogeneous chemical composition and microstructure. In the product described by Zhou, the initial extruded part is driven to a state of macrosegregation by semi-solid forming. As described by Zhou, the microstructure consists of "globular or spherical grains contained in a lower melting alloy matrix." In Column 5, Lines 25 through 45, Zhou describes the evolution of the microstructure and its dependence on the diffusion of low melting elements to grain boundaries and sub-grain boundaries. Therefore, the chemical composition of Zhou's semi-solidly formed material is highly segregated. Variations in chemical composition can have a profound affect on both mechanical properties and corrosion resistance properties.

31. The degree of segregation in Zhou's semi-solidly formed 6061 is evident in both

Figures 11 and 12. In Figure 11, the low melting alloy matrix must be of different chemical composition (see Figure 14). The nominal composition of Zhou's material does fall within the specification for 6061, but the composition of the particles and the low melting matrix are significantly different. As shown in Figure 14, the low melting matrix must be rich in a combination of Mg, Cu, and Si, represented by the vertical blue line. In order for the alloy to abide by the laws of mass conservation, the aluminum-rich particles within the matrix of Zhou's alloy must have an alloy composition less than the nominal value for 6061 (vertical green line), *i.e.*, the vertical red line. In contrast, a typical wrought processed 6061 alloy and a centrifugally cast JC6061-T6 alloy will not have the composite structure of Zhou's material, *i.e.*, two distinct regions of matrix and particles.

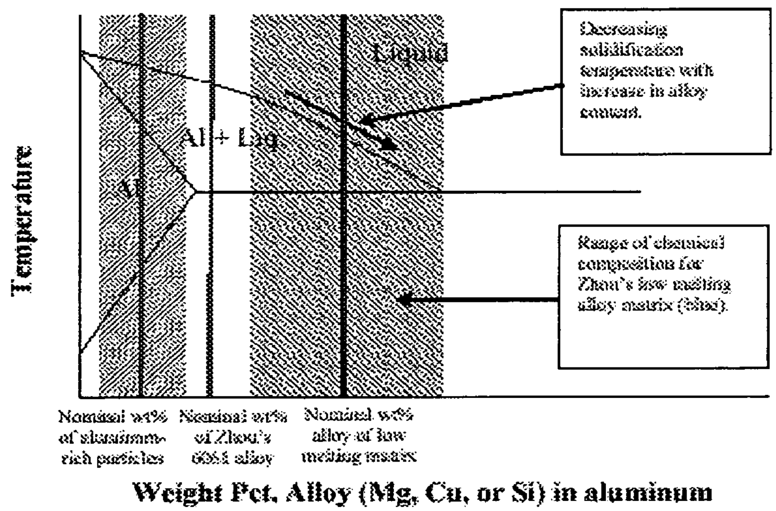


Figure 14. Schematic of the phase diagram for 6061 aluminum illustrating the difference between the nominal chemical composition of Zhou's 6061 alloy and the low-melting alloy matrix.

32. More significantly, these materials will not exhibit the wide range of chemical compositions seen in Zhou's material. For a typical wrought processed 6061 alloy and a centrifugally cast 6061-T6 alloy, a typical range of chemical composition is shown in Figure 15.

The nominal composition is identical to Zhou's material, however, two distinct regions and the associated wide range of chemical compositions will not be present.

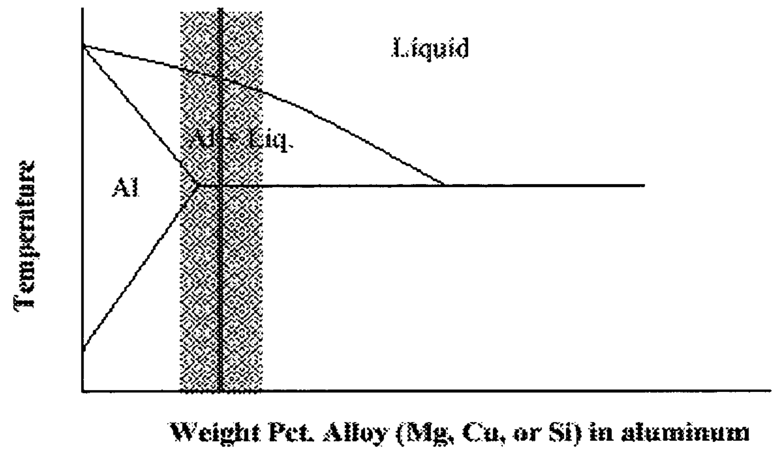


Figure 15. Schematic of the phase diagram for 6061 aluminum for wrought 6061 and centrifugally cast 6061, illustrating the relatively narrow chemical composition range.

33. The presence of lower melting point constituents can have a profound affect on both mechanical and corrosion properties. When lower melting constituents are present, eutectic melting must be addressed in subsequent heat treating operations. One alloy where eutectic melting is a concern is 7075 aluminum. In Figure 16, a centrifugally cast 7075 aluminum alloy was intentionally heat treated above the eutectic melting temperature to demonstrate both the transformation of the microstructure and degradation of mechanical properties. The microstructure consists of grains that are almost completely surrounded by a low melting eutectic structure at the grain boundaries. The microstructure shown in Figure 16 is similar to that of Zhou's material shown in Figure 11. Mechanical properties for the material shown in Figure 16 are as follows: 59 ksi tensile stress, 51 ksi yield stress, and 0.5% elongation. Compared to typical mechanical properties of JC7075-T6 produced by the present method (77 ksi tensile stress, 67 ksi yield stress, and 8% elongation), the material shown in Figure 16 had a decrease in

strength properties by approximately 23% and a decrease in ductility by approximately 94%. Based on these results, the presence of a near-continuous or continuous low melting point second phase matrix can be a severe detriment to the material.

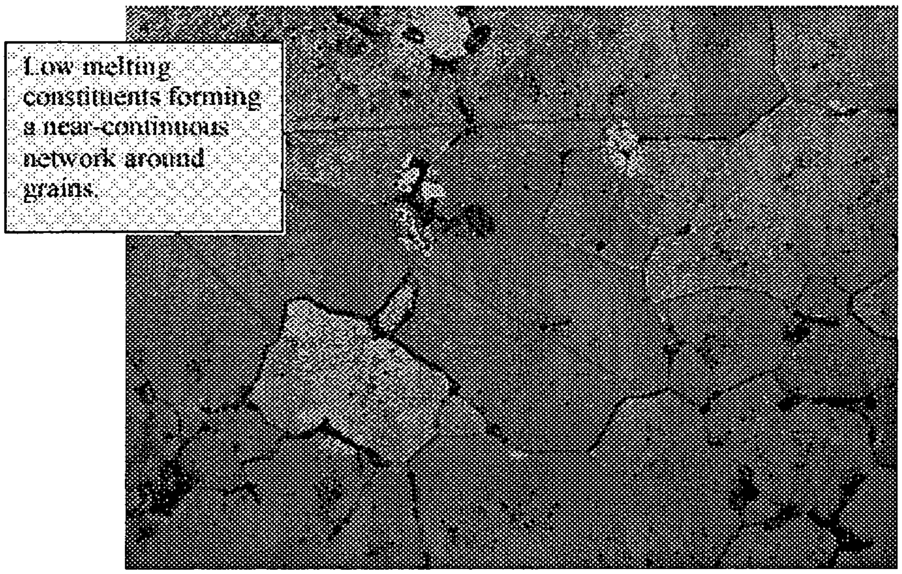


Figure 16. Centrifugally cast JC7075-T6 aluminum exhibiting eutectic melting during solution treatment. Mechanical properties are as follows: 59 ksi (tensile stress), 51 ksi (yield stress), and 0.5% elongation.

34. Zhou claims to have "a product with superior mechanical properties," but fails to produce data that supports the claim and a description of what materials are being compared. Based on the time-temperature profile of Zhou's semi-solidly formed 6061, reproduced as Figure 17, it is apparent that the mechanical properties of the material would not be expected to meet those of wrought 6061 aluminum in the T6 condition. In Stages 10, 11, and 12 of Zhou's process, Figure 17, the material has been strengthened by a combination of work-hardening and precipitation hardening, presumably leaving the material in a T4 condition, *i.e.*, solution treated, quenched, and naturally aged. By heating the material to a temperature significantly higher than the

recrystallization temperature, T_R, all mechanical properties associated with the T4 temper would be removed. Based on Zhou's time-temperature profile shown in Figure 17, one would expect the material to be in the O-temper, *i.e.*, an annealed condition. The fracture mechanism, however, will likely be different from wrought 6061 and centrifugally cast 6061. Since Zhou's material is a composite material of aluminum-rich particles within a low melting matrix, the failure will likely initiate at the weaker of the two materials or the interface between the two phases. Because the interfacial area between the matrix and the particles is large, one would expect the material to contain a high number of potential initiation sites for failure. In comparison, both wrought 6061 and centrifugally cast 6061 contains a relatively small amount of discrete isolated second phase particles, resulting in fewer potential initiation sites for failure.

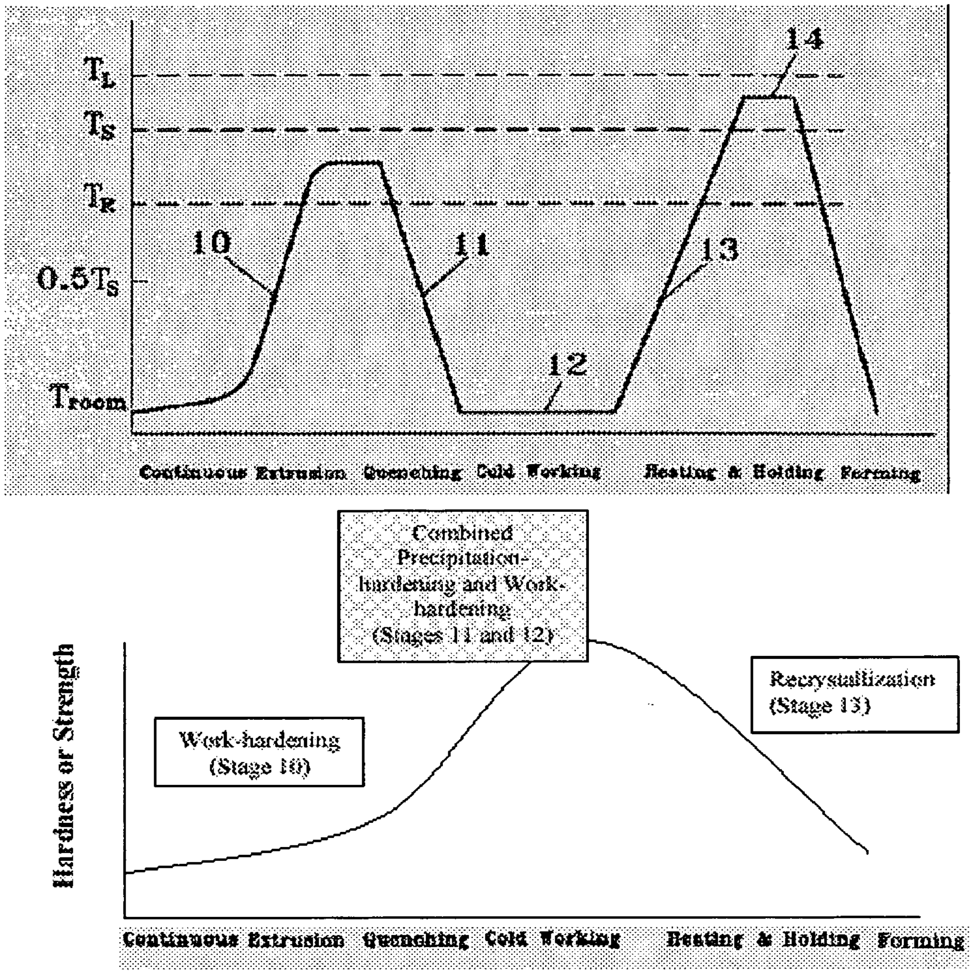


Figure 17. Reproduction of Figure 1 of Zhou (top), illustrating a schematic time-temperature profile in accordance with the process of continuous extrusion with a solid feedstock of Zhou's invention. Bottom schematic represents the anticipated mechanical properties associated with the different stages of Zhou's process.

35. In summary, Zhou's semi-solidly formed 6061 material is significantly different from aluminum alloy materials produced by the present method based on the following points:

1. Zhou's material contains macrosegregation into aluminum-rich particles and a continuous low-melting point matrix. In comparison, the microstructure of JC6061 centrifugally cast aluminum produced by the present method contains discrete isolated 2 nd phase particles.

2. The chemical composition of Zhou's low-melting point matrix is distinctly different from the aluminum-rich particles. This difference can decrease tensile properties, fatigue properties, fracture toughness properties, and corrosion properties.
3. Zhou has not reported any mechanical or corrosion properties. Based on the time-temperature profile of Zhou's process and the reported microstructure, however, the material can be assumed to be in the annealed condition.

36. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements were made with the knowledge that willful, false statements and the like so made are punishable by fine or imprisonment or both, under Section 1001 of Title XVIII of the United States Code and that such willful, false statements may jeopardize the validity of the above-identified application or any patent resulting therefrom.

By ____________________________
Arvin Montes, Ph.D.

Date ____________________________

Microstructures from a JC6061- T6 Aluminum Centrifugal Casting

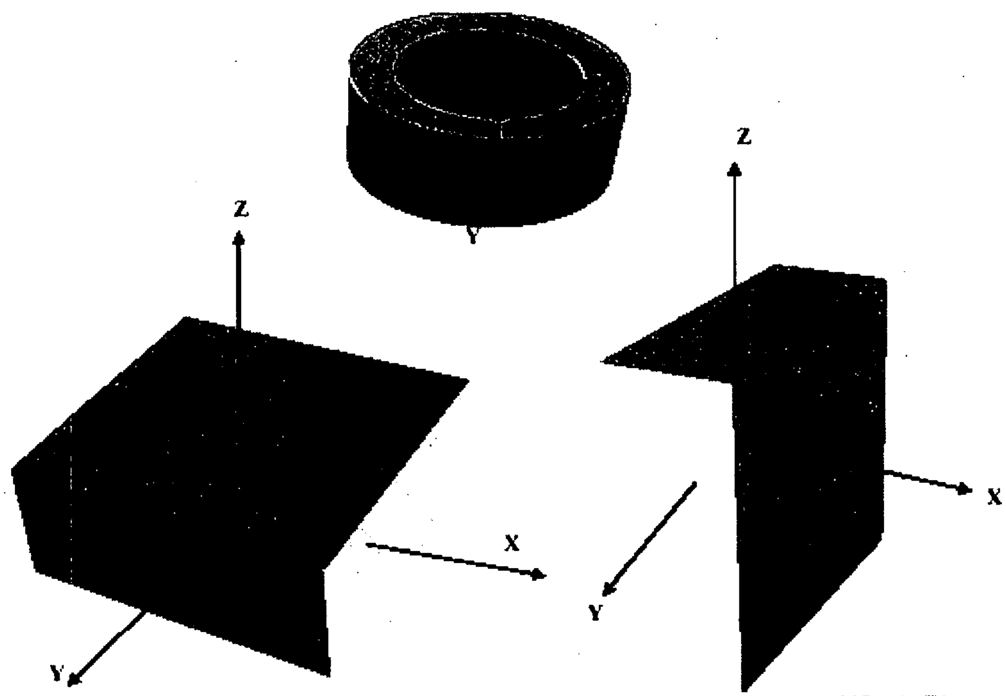


Figure 1. Microstructure of JC6061-T6 in the XY plane.

Figure 2. Microstructure of JC6061-T6 in the YZ plane.

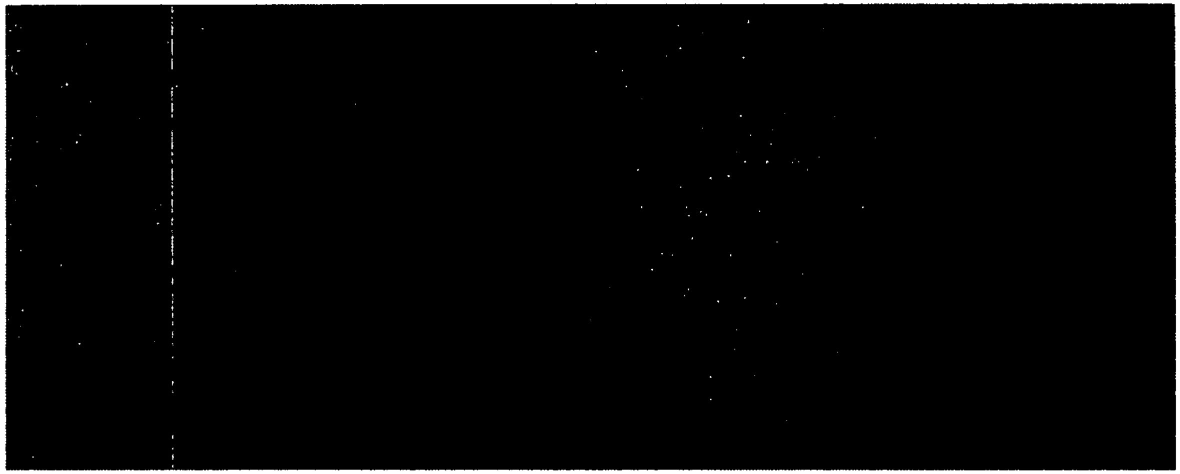


Figure 1. Microstructure of JBM's JC6061- T6 centrifugal casting in the XY plane shown above (left, 100X; right, 200X). Microstructure consists of equiaxed grains with a grain size number of 4 (90μm). Etched with Keller's reagent.

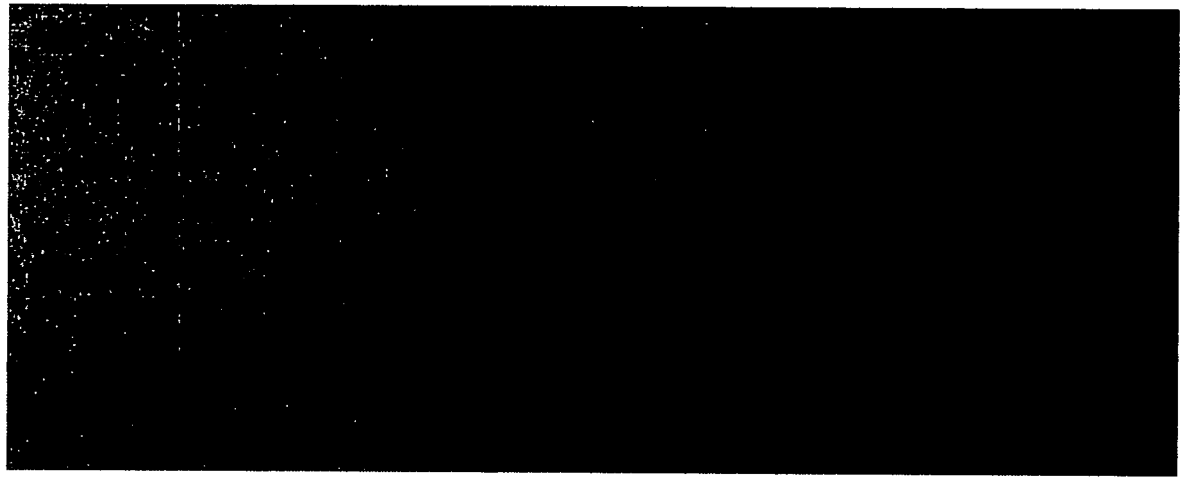
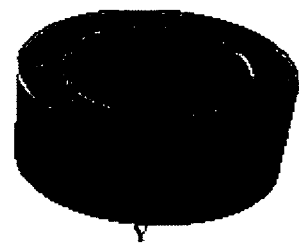


Figure 2. Microstructure of JBM's JC6061- T6 centrifugal casting in the YZ plane shown above (left, 100X; right, 200X). Microstructure consists of equiaxed grains with a grain size number of 4 (90µm). Etched with Keller's reagent.

Microstructures from a JC5252 Aluminum Centrifugal Casting



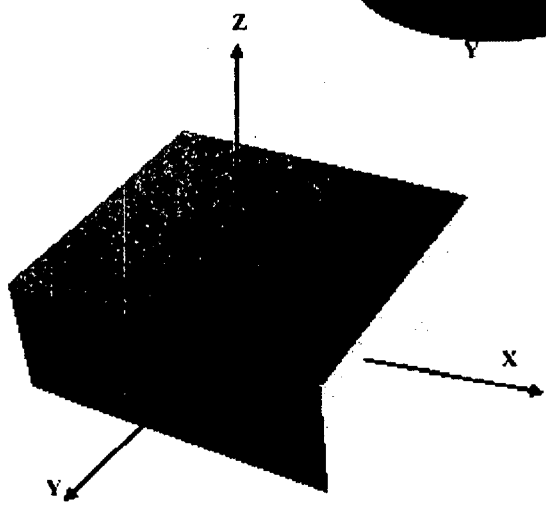


Figure 1. Microstructure of JC5252 in the XY plane.

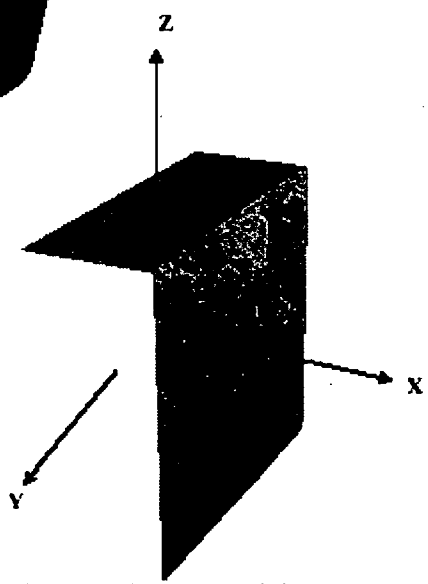


Figure 2. Microstructure of JC5252 in the YZ plane.

Figure 1. Microstructure of JBM's JC5252 centrifugal casting in the XY plane shown above (100X). Microstructure consists of equiaxed grains with a grain size number of 5 (64μm). Etched with Keller's reagent.

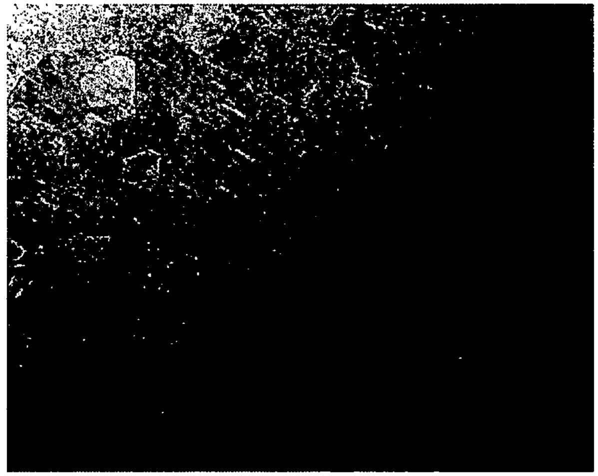


Figure 2. Microstructure of JBM's JC5252 centrifugal casting in the YZ plane shown above (100X). Microstructure consists of equiaxed grains with a grain size number of 5 (64μm). Etched with Keller's reagent.

Microstructures from a JC3003 Aluminum Centrifugal Casting

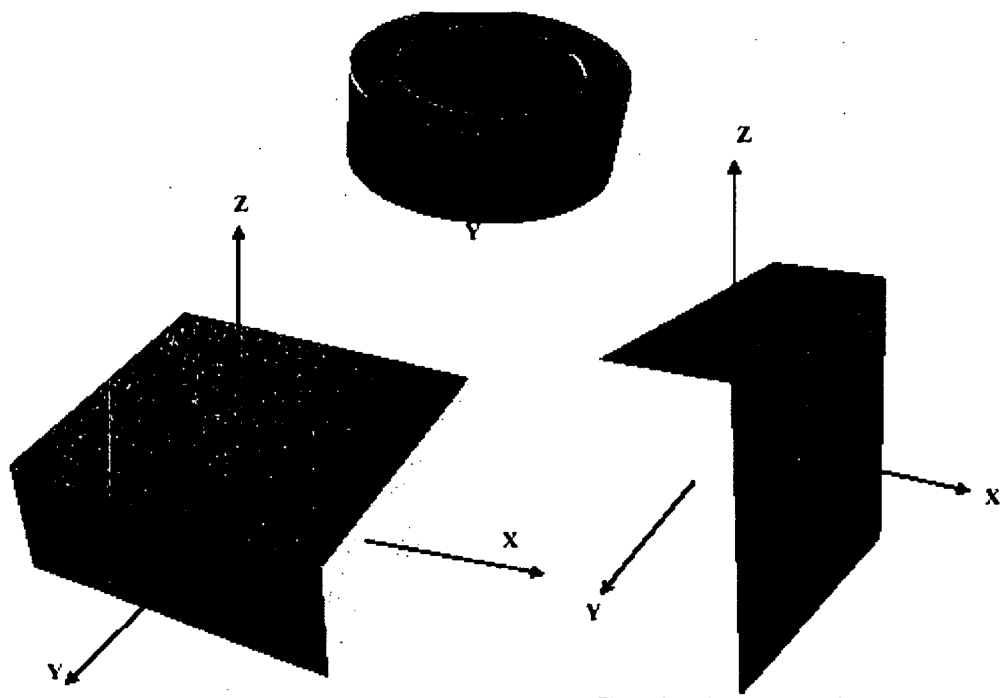


Figure 1. Microstructure of JC3003 in the XY plane.

Figure 2. Microstructure of JC3003 in the YZ plane.

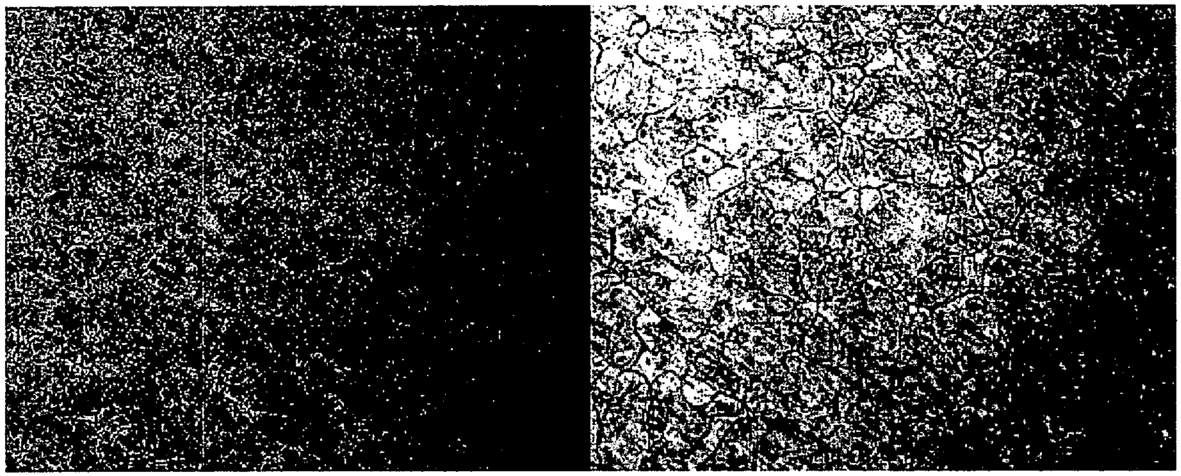


Figure 1. Microstructure of JBM's JC3003 centrifugal casting in the XY plane shown above (left, 50X; right, 100X). Microstructure consists of equiaxed grains with a grain size number of 3.5 (107μm). Etched with Keller's reagent.

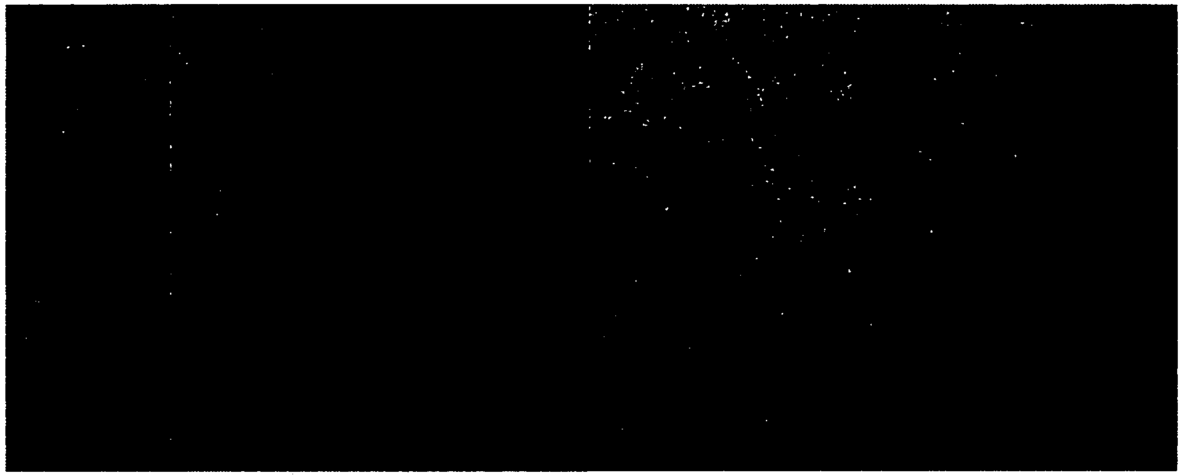


Figure 2. Microstructure of JBM's JC3003 centrifugal casting in the YZ plane shown above (left, 50X; right, 100X). Microstructure consists of equiaxed grains with a grain size number of 3.5 (107μm). Etched with Keller's reagent.

Note: Reference micrographs of wrought 3003 aluminum can be found in ASM Handbook, Volume 9: Metallography and Microstructures, Figures 5 and 6, Page 361. Figures 5 and 6 show the presence of an elongated grain structure after recrystallization. In addition, 2 nd phase particles remain elongated after annealing.

Microstructures from a JC7075-T6 Aluminum Centrifugal Casting

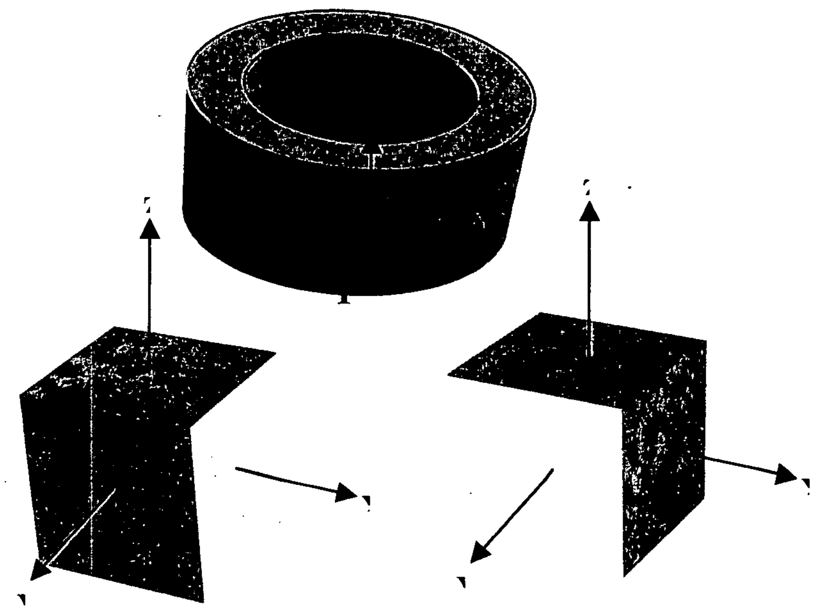


Figure 1.　　Figure 2.

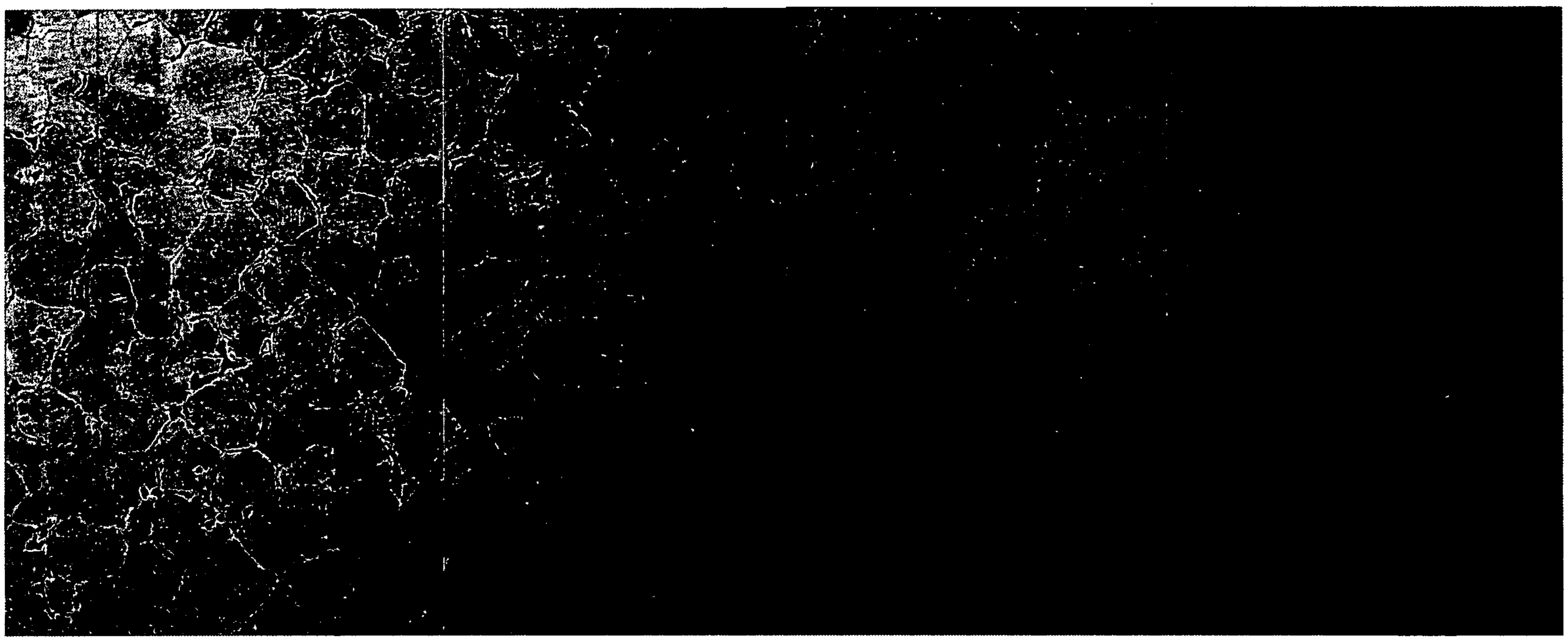
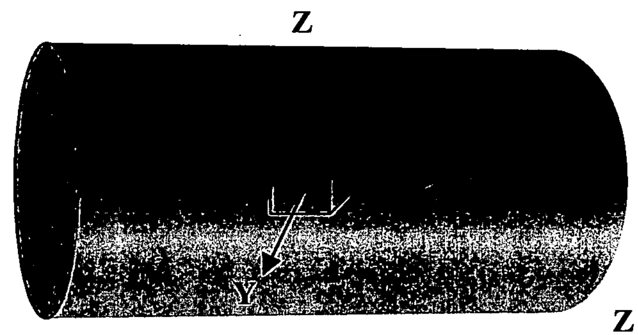


Figure 1. Microstructure of JBM's JC7075-T6 centrifugal casting in the XY plane shown above (left,100X; right, 500X). Microstructure consists of equiaxed grains with a grain size number of 4.5 (75.5μm). Black particles at triple points of grains are 2nd phase particles consisting of Cu, Mg, and Al. Regions at center of grains contains remnants of dendritic structure, i.e., microsegregtion. Etched with Keller's reagent. Mechanical properties are as follows: 77 ksi (tensile), 67 ksi (yield), 7% (elongation).

Figure 2. Microstructure of JBM's JC7075-T6 centrifugal casting in the YZ plane shown above (left,100X; right, 500X). Microstructure consists of equiaxed grains with a grain size number of 4.5 (75.5μm). Black particles at triple points of grains are 2nd phase particles consisting of Cu, Mg, and Al. Etched with Keller's reagent. Mechanical properties are as follows: 76 ksi (tensile), 67 ksi (yield), 6.6% (elongation).

Wrought 7075-T6 Extrusion



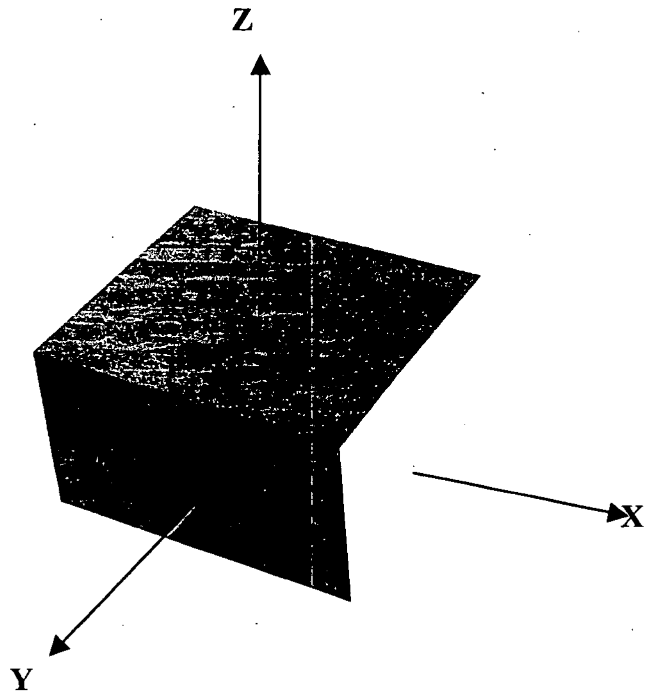


Figure 1. Microstructure of 7075-T6 extrusion in the XY plane.

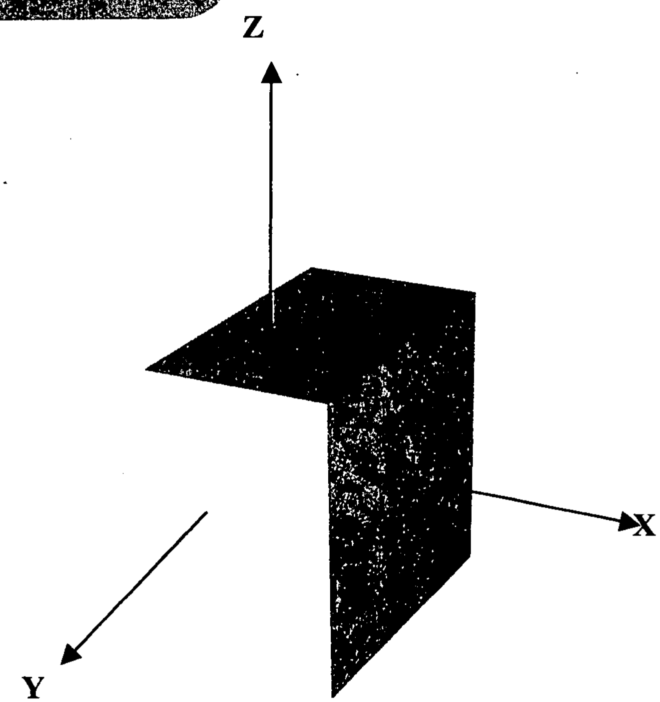


Figure 2. Microstructure of 7075-T6 extrusion in the YZ plane (direction of extrusion).

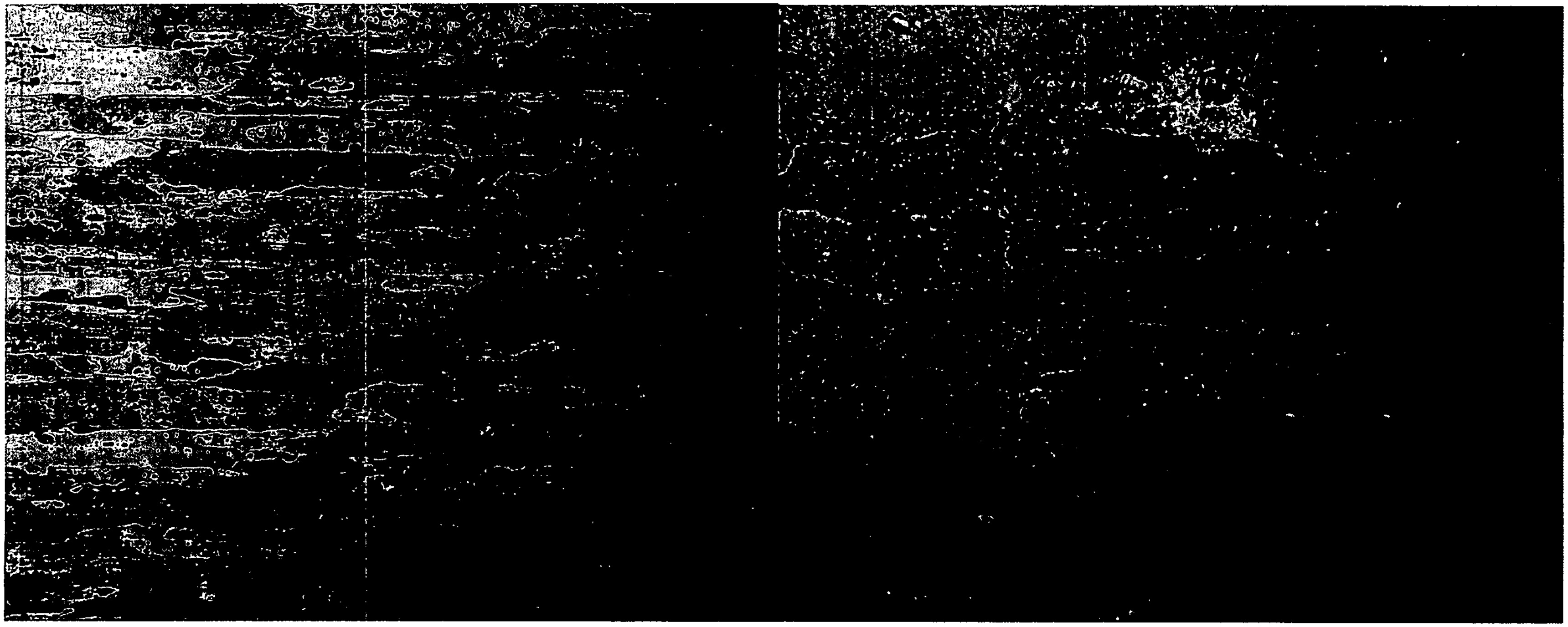


Figure 1. Microstructure of wrought 7075-T6 extrusion in the XY plane, i.e., the direction of extrusion (left, 100X; right, 500X). Elongated grain structure is evident, as well as 2nd phase particles (Al-Cu-Mg). Minimum mechanical properties for extruded 7075-T6 with a diameter between 1.500 and 2.999 inches are as follows: 81 ksi (tensile), 72 ksi (yield), 7% elongation (Aluminum Standards and Data 2000; The Aluminum Association, Inc.)

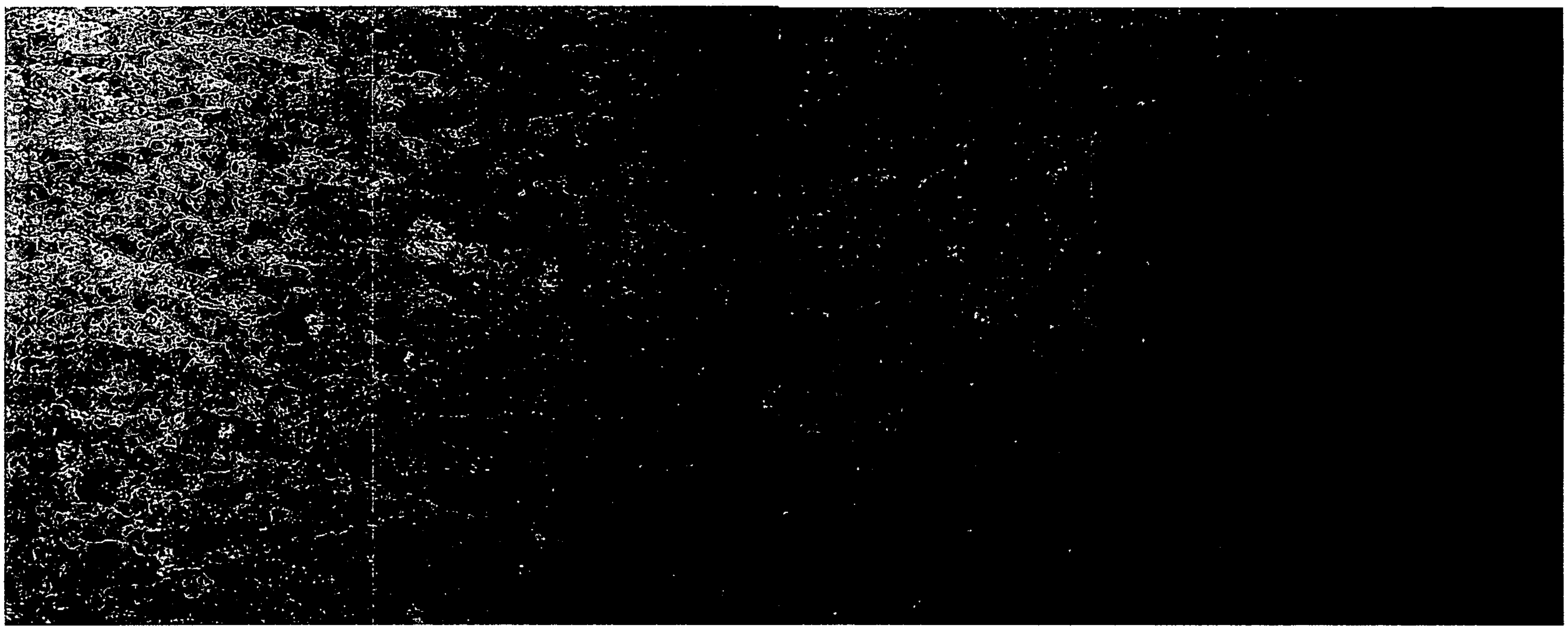


Figure 2. Microstructure of wrought 7075-T6 extrusion in the YZ plane (left, 100X; right, 500X). Black particles are 2^{nd} phase particles (Al-Cu-Mg). Due to the limited stock, mechanical properties were not obtained in this direction.